Neutral and cationic paramagnetic amino-amidinate Iron(II) complexes

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Supporting Information

Neutral and Cationic Paramagnetic Amino-amidinate Fe(II) Complexes; ^{19}F NMR Evidence for Interactions with Weakly Coordinating Anions

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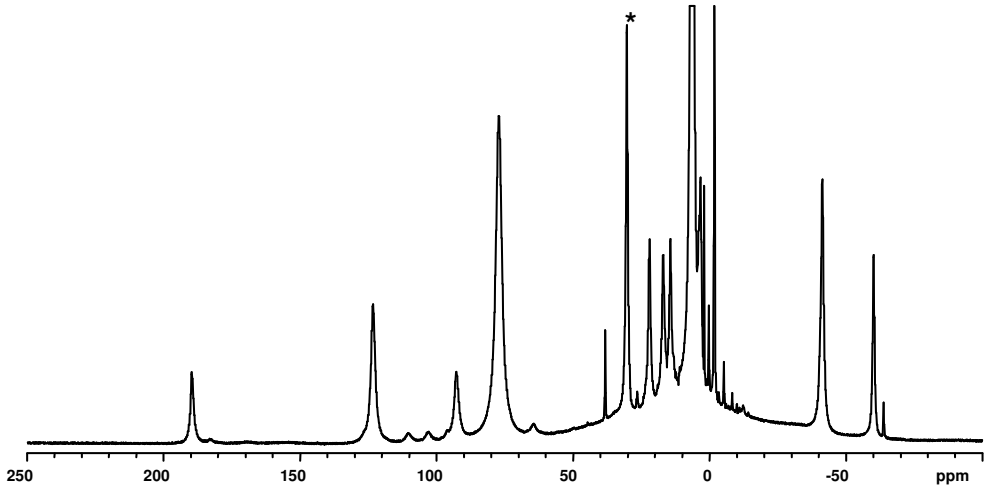

Figure S1 ^{1}H NMR (500 MHz, C_6D_6, RT) spectrum of **4**. Asterisk indicates C_6D_5H

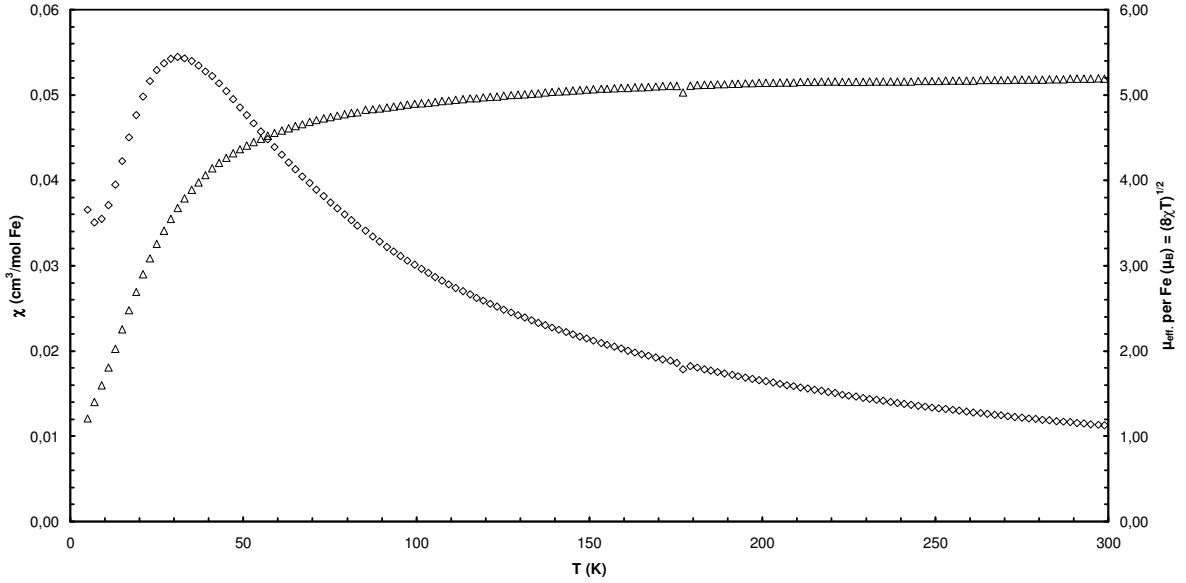

Figure S2 Temperature dependence of magnetic susceptibility and $\mu_{eff.}$ for **1a**.

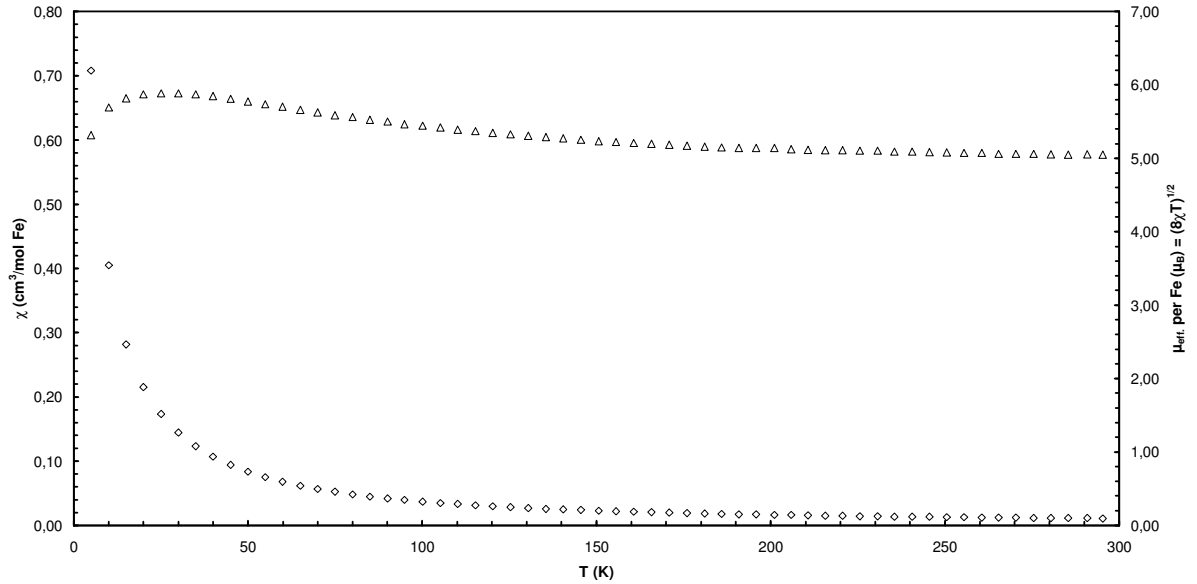

Figure S3 Temperature dependence of magnetic susceptibility and $\mu_{eff.}$ for **1b**.

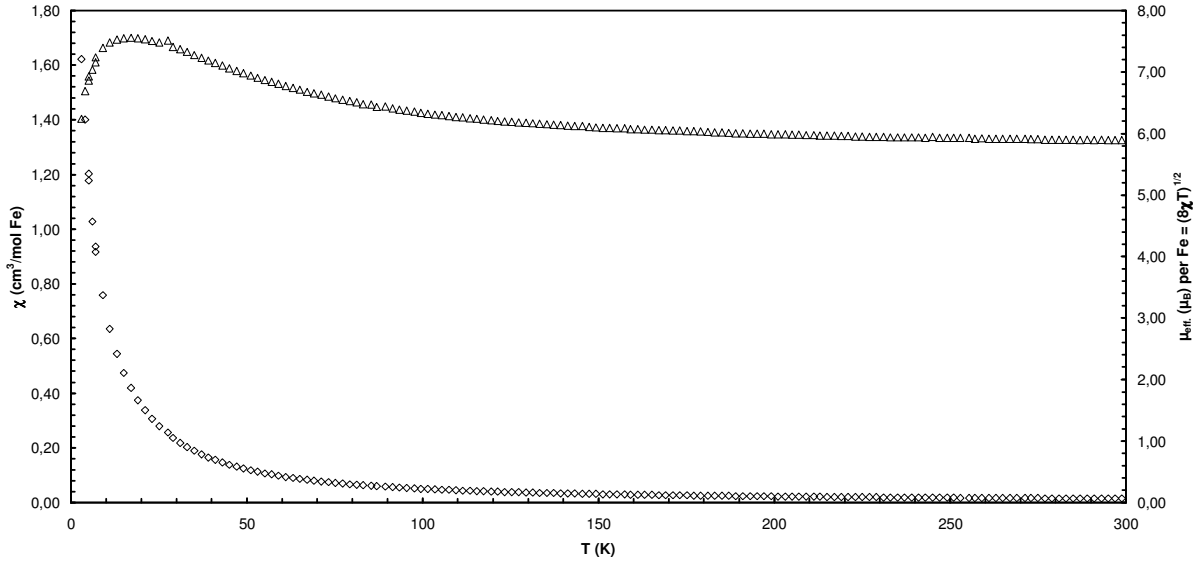

Figure S4 Temperature dependence of magnetic susceptibility and $\mu_{eff.}$ for **4**.

Table S1 Crystal, collection and refinement data for complexes **1a, 2 and 3**.

	1a	**2**	**3**
formula	$C_{46}H_{64}N_6Fe_2Cl_2$	$C_{46}H_{64}N_6Fe$	$C_{26}H_{32}N_3O_3FeCl\cdot 2C_4H_8O$
Fw	883.66	756.90	670.07
cryst. dim. (mm)	0.12 x 0.25 x 0.51	0.44 x 0.19 x 0.17	0.20 x 0.15 x 0.05
colour, habit	yellow, block	yellow, prism	yellow, plate
crystal system	Monoclinic	Orthorhombic	Monoclinic
space group, no.[45]	$P2_1/c$, 14	*Pbca*, 61	$P2_1/n$, 14
A (Å)	14.889(2)	17.6967(8)	9.4467(8)
B (Å)	9.889(1)	18.5086(9)	20.014(2)
C (Å)	16.661(1)	25.923(1)	18.161(2)
A (°)	90	90	90
B (°)	116.03(1)	90	96.305(1)
Γ (°)	90	90	90
Z	2	8	4
V (Å^3)	2204.3(4)	8490.9(7)	3412.9(6)
ρ_{calc} (g/cm^3)	1.331	1.184	1.304
Diffractometer	Enraf Nonius CAD-4F	Bruker Smart APEX CCD	Bruker Smart APEX CCD
Θ range (°)	1.35 – 27.0	2.24 – 27.31	2.33 – 23.74
Λ (Å)	0.71073 (Mo Kα)	0.71073 (Mo Kα)	0.71073 (Mo Kα)
scan mode	φ- and ω-scans		
T (K)	180	100(1)	105(1)
data collect. time (h)	84.3	8.3	27.0
No. of meas. refl.	5266	71048	31758
No. of unique refl.	4802	9731	9004
M (cm^{-1})	8.2	3.94	5.64
No. of parameters	381	734	416
No. of restraints			
weighting scheme: a,b[a]	0.1087, 0.2692	0.0498, 4.7606	0.0945, 0.0
$R(F)$ for $F_0 \geq 4\sigma(F_0)$[b]	0.0482	0.0429	0.0628
$wR(F^2)$[c]	0.1443	0.1094	0.1737
res. el. dens. (e/Å^3)	–0.90, 0.89(11)	–0.46, 0.45(6)	–0.47, 0.8(8)
GoF[d]	1.040	1.036	0.978

[a] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$

[b] $R(F) = \sum (||F_o| - |F_c||) / \sum |F_o|$,

[c] $wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$,

[d] GoF = $[\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{½}$, n = # refl., p = # param. refined.

[e] The conventional space group is $C2/c$.

Transformation: $(a') = (-1\ 0\ -1)(a)$; $(b') = (0\ 1\ 0)(b)$; $(c')_{C2/c} = (1\ 0\ 0)(c)_{I2/a}$ $a' = 29.221$, $b' = 8.132$, $c' = 18.514$ Å, $\beta' = 127.72°$

Table S2 Crystal, collection and refinement data for complexes **1b** and **4**.

	1b	**4**
formula	$C_{28}H_{48}N_6Fe_2Cl_2Si_2$	$C_{42}H_{62}N_6Fe_2Si_2$
Fw	707.50	818.86
cryst. dim. (mm)	0.10 x 0.20 x 0.24	0.22 x 0.19 x 0.17
colour, habit	orange, plate	red, parallelepiped
crystal system	Monoclinic	Monoclinic
Space group, no.[45]	*I*2/*a*[e], 15	*C*2/*c*, 15
a (Å)	18.514(1)	14.9022(9)
b (Å)	8.132(1)	13.1648(8)
c (Å)	23.124(6)	22.578(1)
α (°)	90	90
β (°)	91.58(1)	103.166(1)
γ (°)	90	90
Z	4	4
V (Å^3)	3460.1(10)	4313.0(4)
ρ_{calc} (g/cm^3)	1.350	1.261
Diffractometer	Enraf Nonius CAD-4F	Bruker Smart APEX CCD
θ range (°)	1.39 – 27.0	2.41 – 23.13
λ (Å)	0.71073 (Mo Kα)	0.71073 (Mo Kα)
scan mode	φ- and ω-scans	
T (K)	130	125(1)
data collect. time (h)	127.3	11.9
no. of meas. refl.	7598	20647
no. of unique refl.	3751	5349
μ (cm^{-1})	10.83	7.64
no. of parameters	265	288
no. of restraints	6	2
weighting scheme: a,b[a]	0.0252, 13.57	0.0486, 2.6612
$R(F)$ for $F_0 \geq 4\sigma(F_0)$[b]	0.0514	0.0483
$wR(F^2)$[c]	0.1128	0.1163
res. el. dens. (e/Å^3)	–0.84, 0.81(7)	–0.45, 0.46(7)
GoF[d]	1.069	1.017

[a] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [\max(F_o^2,0) + 2F_c^2] / 3$

[b] $R(F) = \sum (||F_o| - |F_c||) / \sum |F_o|$,

[c] $wR(F^2) = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$,

[d] $\text{GoF} = [\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{½}$, n = # refl., p = # param. refined.

[e] The conventional space group is *C*2/*c*.

Transformation: $(a') = (-1\ \ 0\ \ -1)\ (a)$; $(b') = (0\ \ 1\ \ 0)\ (b)$; $(c')_{C2/c} = (1\ \ 0\ \ 0)\ (c)_{I2/a}$

$a' = 29.221$, $b' = 8.132$, $c' = 18.514$ Å, $\beta' = 127.72°$